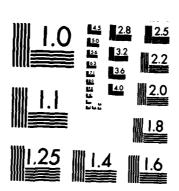
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RADC-TR-84-259
Final Technical Report
December 1984



DOWNDRAW EXTRUSION OF ULETM GLASS

Corning Glass Works

P. M. Smith and C. E. Peters

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Air Force Systems Command
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GLOSSARY OF TERMS AND ABBREVIATIONS

CTE Coefficient of Thermal Expansion

I-100 "Inducto 100" Console Mounted Motor Generator Set

ULETM Ultra Low Expansion Glass

Charge Glass Loaded into Furnace For Remelting

Cyclops Minolta Land "Cyclops" Hand Held Infrared Pyrometer

Pot Setting Potentiometer Setting

1. INTRODUCTION

In September 1982 Corning submitted a proposal to Air Force Systems Commands Rome Air Development Center for conducting a program to evaluate the feasibility of remelting and drawing Code 7971 ULE glass. This proposal was made as part of an effort by Corning to reduce the costs and lead times of lightweight mirror core components. ULE glass struts produced by a downdraw process would reduce costs and manufacturing time through increased material utilization.

The first step in the downdraw program was to demonstrate by laboratory experiments, the basic feasibility of remelting code 7971 glass. The results of this experiment were presented in H.E. Hagy's report "Evaluation of Thermal Expansion and Viscosity Changes in ULE Titanium Silicate Due to Thermal History Simulation of Remelt for Redraw", September 28, 1983. In November, 1983, after the experiment was completed and the results were reviewed by RADC, the program proceeded with the design and installation of a furnace system for remelting and drawing ULE glass. In June, 1984 two trial runs were made and the drawn ULE glass samples were measured to evaluate the effect of the process on the glass thermal expansion.

2. LAB TEST

2.1 Experiment Plan

A laboratory experiment simulating the conditions of remelting ULETM glass in the downdraw furnace system was to be conducted to determine the effect of remelting on CTE.

A piece of ULETM glass, shown to be homogeneous for CTE was to be selected for evaluation. One-half of the material would be remelted in a small induction furnace under conditions similar to those in the proposed downdraw furnace.

This material, after undergoing the time and temperature cycle would provide the center component of a sandwich seal. A sandwich seal consists of 3 layers of glass, a center component of unknown expansion and two outer

components of known expansion. The outer two components would come from the unprocessed half of the original material. A photoelastic analysis (seal test) would provide data on any change in CTE. Remelted material would also be evaluated for change in thermal expansion by dilatometry. In addition strain and anneal points would be determined and compared with unprocessed material.

2.2 Apparatus

The glass remelting operation was conducted in the Developmental Melting Facility at Sullivan Park, Corning, N. Y.

An Inducto 30 console mounted motor generator set was used as the induction coil power supply. A concentric arrangement consisting of a fused silica outer sleeve, graphite felt, and carbon crucible/susceptor was used to simulate the conditions to be found in the full scale furnace.

CTE results are obtained with a Friedel polarimeter and with a dilatometer.

Strain point and anneal point are determined with a beam bending viscosimeter.

2.3 Procedure

A laboratory experiment was conducted to determine if remelting ULE TM glass effected the coefficient of thermal expansion. Three 2" dia. x 4" long cylindrical slugs were core drilled from a block of ULE glass 6" x 6" x 4". Each slug was heated in an induction furnace and held for 1/2 hour at 1650°C to simulate conditions for remelting and drawing. Following these remelting thermal cycles, all three slugs were annealed together using the standard anneal for ULE TM.

A 1/8" x 3 7/8" x 1 7/8" piece was cut from each of the three slugs. Each of these pieces were fritted to two identical parts from the original block to form a symmetrical sandwich seal.

A 3/8" x 4" diameter rod was cut from each of the three slugs and from the original block. These pieces were used for determination of the CTE with the superdilatometer.

A 1/10" x 1/10" x 3" beam was cut from each of the three slugs and from the original block. These pieces were used to determine this annealing point and the strain point using beam bending viscometry.

2.4 Results

2.4.1 Symmetrical Sandwich Seal

Slug No.	Block Minus Slug (ppB/°C)
1	+0.36
2	+4.65
3	-0.90

2.4.2 Superdilatometer

Slug No.	Block Minus Slug (ppB/°C)
1	+ 2
<u> </u>	+18

2.4.3 Beam Bending Viscometry

Slug No.	Annealing Point ^O C	Strain Point °C
1	1008	897
2	1008	898
3	1009	899
Original	1007	896

2.5 Conclusions

Evaluation of the results obtained with sandwich seals and the superdilatometer show no significant changes in thermal expansion are caused by the simulated remelt thermal cycle. Slug No. 2 does show a small expansion difference which could have existed prior to remelting.

The anneal point and strain point show no significant differences. This indicated that material composition and structure are unchanged. None of the results found in this laboratory simulation indicate that the CTE of ULE TM glass will change during a remelt process.

3. DEVELOPMENT PROGRAM

3.1 Development Plan

<u>3.1.1</u> Objective

The objective of this project was to demonstrate the feasibility of drawing Code 7971 ULE glass by a process which can be used to make the core material for lightweight mirrors.

In order to accomplish this objective ULETM glass would be remelted and approximately 100 feet would be drawn. Glass property measurements would be evaluated to determine the technical feasibility of the process.

3.1.2 Activities

As part of this feasibility demonstration, equipment that would be suitable for use during the process development trials would be developed. Engineering drawings of the downdraw system would be prepared including equipment design, facility layout and services. Materials would be specified and installation of the system would be completed. The equipment would be fully tested and evaluated prior to the start of the process development trials. The fundamental design of this equipment would be similar to that used in the past by Corning to manufacture fused quartz tubing.

3.1.3 Design Concept

This design concept uses a high frequency motor generator set to supply power to a furnace consisting of a water cooled induction coil

surrounding an insulated cylindrical carbon susceptor. ULE glass in a carbon crucible located in the center of the furnace, would be heated to a point where it could be pulled through an appropriately sized orifice in the bottom of the crucible. This design concept is illustrated on p. 23, sec. 4.1

3.2 Equipment Procurement

3.2.1 Motor Generator

An Inducto 100 console mounted motor generator set was obtained from another Corning facility where it had been in storage since 1980. The Inducto 100 unit was manufactured by Inductotherm Corporation for Corning Glass Works in May 1965. It was used as the power source for a quartz crucible manufacturing operation in Corning, N.Y. from 1965 through 1980. The Inducto 100 unit contains the following items:

- Westinghouse High Frequency Motor Generator Serial No. 35-65.
 - Generator 100 KVA, 3000 cycle, 400/800 volts, single phase.

Motor 150 h.p., 220/480 volts, 3 phase.

- High frequency water cooled capacitors for matching coil to power supply.
- Tap switches for impedance matching transformer and capacitor tuning.
- Meters for generator voltage, generator current, output kW, power factor, coil voltage, coil current.
- Variable autotransformer for setting field voltage excitation.
- Circuit fault monitor panel for cabinet water pressure, generator air temperature, starter interlock, generator overload, gas pressure,

clear .00066 weight % Ti_2O_3 dark .0264 weight % Ti_2O_3

Run 2 A plot of temperature versus time is given on p. 60, sec. 4.7. The plate averaged 60° C cooler than the glass as read by an optical pyrometer until 1525° C.

Glass temperature on top of the charge (read through the sight port with an optical pyrometer) was 165°C hotter than the glass temperature orifice at 1625°C. (p. 61, sec. 4.7.1). There was fairly good agreement between the optical pyrometer and the cyclops up to 1500°C. Throughout the run the cyclops averaged 27°C hotter than the optical pyrometer. (p. 62, sec. 4.7.2).

As seen in Run 1 when the generator voltage % is increased, the power (kilowatts) increases. This is illustrated on p. 63, sec. 4.7.3.

Product data is given on p. 64-66, sec. 4.7.4. The table lists information on sample number, sample time, weight, length, pull machine setting, diameter, attenuation and a product description.

A total of 21 lb or 68 feet of clear product was drawn. An additional 45 lb or 262 feet of other product was drawn from the furnace. 4 lb remained in the crucible at the end of the draw. 6.7% of the charge was unaccounted for. average sample had a diameter of .534" and an attenuation of 3.4. Average mass throughput was 20 1b/hour. Throughout the pull, the pull rate was increased in order to determine the effect on the product. Increasing the potentiometer setting on the pull machine results in an increase to the inches per minute of draw as illustrated on p. 67, sec. 4.7.5. This rate increase was linear to a pot setting of 50%. As the product degraded, the rate leveled and then declined (at settings of 50, 60 and 70.) Values for the pull rate at each setting were obtained by averaging the rate obtained using two methods:

 \underline{A} . Periodically timing the product as it

when opened. Other carbon components were reusable.

Run 2 Only 4 lb of glass remained in the crucible at shutdown. This 4 lb was dark and foamy. This crucible also had a crack, one smaller in magnitude than in run 1. The spacer ring and orifice was again plugged. Other components were reusable.

3.5.3 Results

Run 1 The ability to flow ULETM was established. The glass had a working viscosity range when heated sufficiently.

This run demonstrated a need to reconfigure the muffle region to get more cooling. This was done for the next run by increasing the muffle plate ID from 5" to 7".

From the cane obtained during the run, the diameter of the product varied between .070" and .300" or an attenuation range of 2.5 to 10. The average product diameter was .136" or an attenuation of 5.5 (orifice diameter product diameter = attenuation, .75 .136 = 5.5). This demonstrated a need to increase the orifice diameter to 1 1/2" to 2". An orifice of 1 3/4" diameter was used for run 2.

Absolute radial CTE was read on chucks of glass cut from the clear glass remaining in the crucible at shutdown. Two readings of -5 ppb and + 2 ppb were obtained. This is comparable to the - 8 ppb to + 7 ppb radial reading range of the feedstock placed in the furnace, indicating that the heated and cooled clear glass is still ULE Th.

Electron paramagnetic resonance (EPR) spectrum was run on two samples of the glass remaining in the crucible: one clear and one dark and foamy. In both samples reduced titanium (Ti⁻¹) was detected, but the dark sample had a concentration 40 times greater than the clear sample:

temperatures at several locations in the muffle region were recorded. These are given on p. 57, sec. 4.6.3

Run 2 Pull was established at 1650°C by fusing a quartz rod to the side of the plug and tugging. A pair of tongs was also used to help draw the glass to the rollers.

Glass was cut-off below the rollers by two operators - one would hold the cane in place while the second broke the product with a pair of hammers. Samples were labeled with a sequential number and a time at cut-off. Throughout the pull the pull machine rate was varied to determine the effect on the product. One and a half hours into the pull the product changed from clear cane to ware with a dark streak. As the run progressed the product quality degraded. (The pull is discussed further under "Results"). Total run time was 20.3 hours, including a 3.5 hour pull. Temperature profile in the muffle region of the furnace is given on p. 58, sec. 4.6.4.

- \underline{D} . Shutdown followed the procedure listed on \overline{p} . 59, sec. 4.6.5.
- E. Furnace Inspection After the furnace cooled, the water was shut off and the top plate was removed. Felt and carbon components were inspected for wear.

Run 1 Felt near the crucible top was stiff and had a ring of white around the spacer ring. Spacer ring was plugged with ash. Crucible cover looked good. Crucible was cracked at the base. Orifice hole was plugged with glass. The glass remaining in the crucible was dark and foamy. When wire sawed into sections the glass has swirls of blue streaks and bubbles through the body. The bottom 4" of thin glass was clear and had only a few streaks. The top 4" was foamy and bubbly. The furnace had a sulfur-like smell

cyclops pyrometer was also used to gather temperature readings. Temperature of the glass and on the orifice plate was recorded. Log sheets for both runs are attached on p. 48-56, sec. 4.6.2.

The voltage percentage was increased to maintain the temperature heat-up schedule, resulting in a corresponding increase in kilowatts and a gradual decrease to the power factor. Water pressure was maintained at greater than or equal to 20 lb inlet. Inlet temperature was as delivered from the plant system, run 1 between 60° to 65°F and run 2 between 70° to 75°F. Outlet water temperatures increased as the power to the coil was increased, but was kept at less than or equal to 90°F by increasing the water flow to a hot component. Gas inlet pressure was set at 10 psi. Gas flows ran at 10 l/min. through the furnace run.

C. The Pull The "pull" refers to the period of time when glass is drawn from the furnace. The "plug" is the initial section of glass that appears in the orifice opening.

Run 1 At 1500°C (approximately 5 am on 6/6/84), glass could be seen slumping through the orifice. By 1650°C (8 am) glass appeared to be in a workable range, suitable for pulling. Attempts were made to start the pull by fusing a fused quartz rod to the plug and tugging on the rod to draw it through the pull machine rollers. A pull was established three times but could not be maintained. The furnace was shutdown with less than 3 feet of product draw. Total run time was 24.5 hours.

While glass could be drawn through the orifice, attenuation was so great in the muffle region that the product would break before a stabilized pull could be established. The glass that was drawn had a dark surface coating, believed to be the result of getting the furnace too hot. Prior to shutdown,

- 21. Insert carbon gas pin into top plate plug.
- 22. Set o-ring on top plate and seat plug on top.
- 23. Clamp plug in place.
- 24. Connect water lines.
- 25. Connect gas lines.

Run 1 was configured with a 3/4" diameter orifice and a 5" inner diameter muffle plate. Run 2 was configured with a 1 3/4" diameter orifice and a 7" inner diameter muffle plate.

E. Glass Loading After removing the plastic and tissue paper from the cleaned feedstock glass, the disks were lowered into the crucible and centered onto the orifice plate using hand vacuum cups. Load order, charge weight and charge height are given on p. 42, sec. 4.5.

3.5.2 Procedures

- A. Start Up Procedure is given on page 46, sec. $\frac{4.6}{4.6}$
- Furnace Run Run 1 and Run 2 followed a 20.5 hour and 16 hour heat up cycle to 1650°C, respectively. Cycles are listed on p. 47, sec. 4.6.1. Run 1 used nitrogen as a purge Run 2 used nitrogen to 600°C, then switched over to argon (at pressure of 10 psi and flows of 10 1/min). The following parameters were monitored and recorded at 15 minute intervals through the heat-up cycle: furnace temperature, generator voltage percentage, kilowatts, power factor rating, inlet water pressure and temperature, outlet water temperatures (for plates, generator, coil and electrical components), inert gas pressure and flow. Temperature was monitored to 800°C using a type K thermocouple inserted through the orifice and touching the base of the glass charge. At 800°C the thermocouple was removed and temperature was monitored with an optical pyrometer. During run 2 a Land

D. Furnace Assembly

The furnace assembly procedure is as follows:

Install top support plate on tower.

2. Set coil on bottom plate.

- 3. Wrap susceptor with graphite felt.
- 4. Lower silica sleeve onto susceptor assembly.
- 5. Place gas distribution tube on support plate.
- 6. Place gasket on bottom support plate.
- Lower susceptor and silica sleeve onto gasket and support plate.
- 8. Place gas distribution tube, alumina sleeve, muffle plate, support ring, orifice plate on middle support plate.
- 9. Insert assembly into bottom of furnace and bolt middle support plate to top support plate.
- 10. Lower crucible into furnace and seat on orifice plate.
- 11. Place inner alumina muffle and gas distribution tube on bottom support plate.
- 12. Insert assembly into bottom of furnace and bolt bottom support plate to middle support plate.
- 13. Vacuum furnace to remove loose carbon, etc.
- 14. Place crucible cover on top of susceptor.
- 15. Cut felt and layer on top of crucible cover to top of furnace.
- 16. Place gasket on top of silica sleeve.
- 17. Install support rods from bottom plate.
- 18. Install bushings on support rods.
- 19. Set top plate onto support rods -- seat against gasket.
- 20. Install isolation bushings and bolt top plate into place.

- 34-109. For each run three 9.5" diameter disks were prepared using the following steps:
 - Hex oversize per layout lines. <u>l</u>.
 - 2. Circle to specified diameter $(9.5 \pm 1/8")$.
 - Skim top and bottom with 320 grit to <u>3</u>. flat and parallel. Dimension to maximum, but less than 5.1".
 - Hand-bevel to break sharp edges.

Actual dimensions and finished weights are given on p. 42, sec. 4.5 for runs 1 and 2.

- CTE Radial absolute coefficient of thermal expansion was read at one inch intervals on three axes, 120° apart for each feed stock disk. Axial absolute expansion was taken at 1/4" intervals from a standard block corresponding to the same boule (and of the same thickness) as the feedstock for each run. See p. 43, sec. 4.5.1. CTE data is given on p. 44 and 45, sec. 4.5.2.
- Cleaning Feedstock glass was thoroughly cleaned to prevent devitrification from oils and other surface contamination using the following procedure:
 - Wipe with xylene.
 - Rinse with deionized water.
 - Scrub with powdered detergent and deionized water.
 - Rinse with deionized water.
 - Swab with muriatic acid. Let acid set for 10 minutes.
 - Rinse with deionized water.
 - <u>6</u>. Wipe dry with paper towel. (Handle clean ware with white cotton gloves).
 - 8. Wrap clean ware in tissue, then plastic. Seal plastic with masking tape.

following parameters were monitored and recorded at 15 minute intervals through the heat up cycle: furnace temperature, generator voltage percent, output kilowatts, power factor, inlet water pressure and temperature, outlet water temperatures, purge gas pressure and flow rates. Furnace temperature was monitored to 800°C with a type K thermocouple inserted through the orifice into the furnace cavity approximately 2 inches. At 800°C the thermocouple was removed and the orifice plate temperature was monitored with an optical pyrometer.

A heat-up schedule was maintained by observing furnace temperature and manually varying the power to the coil. A plot of the actual heat-up schedule is shown on p. 36, sec. 4.4. A log sheet from the dry run is included on p. 37-41, sec. 4.4.1.

3.4.1 Debugging and Operational Results

Operation of the induction remelt system during the dry run was remarkably smooth and trouble free. The major problems encountered were associated with the cooling water system. Supply pressure variations caused shutdowns of the motor generator set early in the run. Pressure switch was reset and no further problems were encountered. During the run, excessive condensation of the external surfaces of the cooling lines was evident. This problem was solved by insulating all cooling lines.

After the run was completed, the furnace was disassembled and inspected. Oxidation of the carbon components was minimal. No problem conditions were observed on any of the other furnace components.

3.5 Glass Remelting and Drawing Operation

3.5.1 Set Up

A. Glass Preparation - Finishing The glass for run 1 was obtained from a boule identified as 94-XXX, glass remaining from a ring seal segment. Glass for run 2 was from boule

was installed on the furnace supply to automatically shutdown the furnace in the event cooling water flow was interrupted. A pressure gauge and thermometer were installed in the supply line for monitoring incoming water pressure and temperature. Rubber hose was used to connect from the main supply to the water cooled components. All lines were insulated to prevent condensation. Return lines were piped to a sight drain where flow could be monitored visually. Thermometers in the return water stream provided return water temperature data. Water was piped from the sight drains to an existing floor drain.

C. Purge Gas

Two gases were supplied to the furnace for purging. Nitrogen was delivered from an existing Linde 93VCC liquid nitrogen storage tank located outside the plant. The nitrogen was piped through a filter located ahead of the gas distribution and control panel. Argon was supplied from Linde GP-45 Liquid Storage Cylinders. Argon was also filtered prior to delivery to the gas distribution and control panel.

3.3.2 Equipment Installation

Installation of all equipment and services was by Canton plant's Trades and Electrical personnel. Existing installation tooling and equipment were adequate for completion of the project.

3.4 Equipment Start-Up

A "dry" run was conducted to evaluate the operation of the furnace system prior to remelting and drawing glass. The motor generator was brought up to speed and power was applied to the induction coil. System capacitance was varied in order to obtain the best possible loading. A fully assembled furnace, minus the glass charge, was heated to 1800°C over a 28 hour heat-up cycle. Nitrogen was used to purge the furnace throughout the run. The

normal force (i.e. = 1). This force could vary between 0 and 50 pounds.

3.3 Installation

ANALYSIS A SAMPLEY CONTROL

3.3.1 Facility and Process Services

A. Electrical Power

Power was required for operation of the motor generator set, pull-machine, and miscellaneous electrical equipment.

The motor-generator set requires power for a 150 HP, 480V, 3 phase AC motor. A Westinghouse fusible plug-in switch (model TAP-365) rated at 400 amps was installed in bus duct No. 4 (600A, 480V, 30, 3W). The switch was fused with 3-225 amp dual element fuses in order to provide backup overload protection. Power was supplied to a fused disconnect switch positioned in a location visible and accessible from the motor generator set. The power was then supplied to the reduced voltage motor-starter controlling the motor.

Power for the pull machine and miscellaneous electrical equipment was supplied from existing circuits located in the furnace area.

B. Cooling Water

Water was required for cooling the motor generator set, capacitors, high frequency transformer, induction coil and furnace components. A connection was made to the existing plant process water system and piped to the furnace area. A cartridge type filter unit, capable of filtering 100 micron particles, was installed in line ahead of water cooled equipment. Separate supplies were provided from a 1 1/2" main supply line to individual furnace components. Manually controlled needle valves were used to regulate the flow to the equipment. A pressure switch

furnace. Gas was introduced into the bottom of the furnace through a stainless steel manifold located between the inner and outer alumina cylinder, the outer alumina cylinder and susceptor, and between the susceptor and fused silica shell. Gas was delivered into the crucible through a carbon tube which extended from the center top plate through a hole in the top of the crucible.

E. Orifice

The orifice was designed for forming round cane. A round hole was drilled in a carbon plate tapered down from the outside diameter to the hole. The orifice is illustrated on p. 35, sec. 4.3.1.

3.2.3 Pull Machine Design

A detailed design for a downdraw type pull machine was provided by Corning's developmental melting department. Machines of the same design were used by this department in a development facility for pulling glass cane and strip.

A gear driven pair of rollers, rotating in opposite directions, is mounted approximately 6 - 7 ft. below the furnace so that glass can be fed between the rollers and pulled continuously. A variable speed motor with a range of 300 to 1800 RPM combined with a 300:1 speed reduction through gearing provided a 1 RPM to 6 RPM roller speed. With the 4" diameter rollers that were used, linear pull rate could be varied from 12.5 in./min. to 75 in./min.

Pulling force could be varied by regulating the pressure supplied to an air cylinder attached to a roller and lever arm assembly. An increase in pressure would increase the force applied at one end of a lever arm thus increasing the force applied normal to the glass by the roller which was mounted at the opposite end of the lever. The pull force was assumed to be approximately equal to the

induction coil, fused silica outer shell, layered graphite felt blanket, and carbon susceptor. Centered in this furnace is a stacked arrangement which consists of a carbon crucible, carbon orifice plate, carbon support ring, carbon muffle plate and alumina cylinder.

Design of this stacked arrangement is such that by varying the height and/or diameter of various components the cooling rate of the drawn glass can be affected. All furnace components are contained between water cooled stainless steel plates on the top and bottom. Three stainless steel plates provide support for the furnace assembly. An outer plate supports the fused silica sleeve, layered graphite felt and carbon susceptor. A middle plate attaches the outer plate supports, the alumina ring, muffle plate, support ring, orifice plate and crucible assembly. An inner plate attached to the middle plate supports a high density alumina cylinder which is also used to control the cooling rate of drawn glass. Specifications of all components are included on pp. 27-34, sec. 4.3.

C. Glass Loading

A heavy duty single cup vacuum lifter was purchased for use in loading the glass into the furnace. Loading would be done through the top of the furnace prior to heating.

D. Purge Gas

A purge gas system was required to remove oxygen from the furnace cavity, preventing degradation of the carbon components due to oxidation at high temperatures. The system was designed for use with either nitrogen or argon as a purge gas. Purge gas was delivered from a pressurized liquid gas cylinder to a flow control panel consisting of a pressure regulator and a bank of flow meters for control of gas flow to various parts of the

production of 2200 pounds of finished ULE glass strip 6.62" wide x (.050" - .100") thick x 25" long.

- Design an induction remelt furnace system using the I-100 motor generator set as the source of power. The furnace must be capable of remelting ULE glass for downdraw into strip form. The furnace will be designed to provide maximum possible charge and strip size.
- Develop a method for preparing and loading ULE glass feedstock for remelting.
- Specify and design a purge gas system for the induction furnace.
- Determine orifice design requirements and design orifice for downdraw of ULE^{IM} glass. Determine methods of establishing glass flow.
- Assist plant personnel with installation and startup.
- Provide sufficient documentation and drawings to allow the Canton Plant to install and operate equipment.

Due to the developmental nature of this project an accurate evaluation of the capabilities of this system to produce strip of the size required was impossible. The evaluation indicated that this system did have the capability of producing strip in sufficient sizes and quantities to provide the process information that would be necessary for design of a production size furnace.

B. Furnace

The furnace was designed using the I-100 as the source of power. The furnace consists of a concentric arrangement of a water cooled furnace water pressure and system ground detection.

- Audible alarm for major fault indication.

Repair records showed that the Westinghouse Motor Generator received a major overhaul shortly before it was removed from service in 1980. The Inducto 100 was installed in the Canton Plant and tested to determine what repairs or modifications were necessary. The test included a general inspection of the unit by Engineers from Inductotherm Corp. and Corning. A summary of actions and recommendations is included on pp. 24-26, sec. 4.2

Operation of the Motor Generator Set was smooth and vibration free. No problems were experienced during this test. Furnace controls were operational. Control panel meter and the ground detector were defective. Cooling water hosing was deteriorated. The capacitance of the I-100 was not sufficient to match the inductance of the coil which was used for the test. The following actions were taken to refurbish the I-100 unit.

- A. All control panel meters were replaced.
- B. Capacitors replaced.
- C. Water cooling hosing replaced.
- $\underline{\mathbf{D}}$. Cooling water pressure switch replaced.

3.2.2 Furnace Design

A. Scope

Corning's Furnace Engineering Department provided detailed design for the induction remelt furnace. Prior to starting detailed design work, a scope of work to be done by furnace engineering was outlined as follows:

- Evaluate the capability of the system for

came through the rollers.

 \underline{B} . Calculating the rate for each piece from one sample to the next:

Length of Piece 2 Elapsed time between piece 1 and piece 2

Pull rate at each pot setting is given on p. 68, sec. 4.7.6.

With a change in the pull rate effects on the product are evident. As the pull rate (inches per minute) increases the following linear effects are noted:

Weight per inch of product decreases (p. 69, sec. 4.7.7).

Diameter decreases (p. 70, sec. 4.7.8).

Attenuation increases (p. 71, sec. 4.7.9).

Mass throughput (in pounds per hour) can be determined by using the formula M = 60 PD where M= mass throughput in pounds per hour, P = pull rate in inches per minutes, D = Cane "density" in pounds per inch. In general mass throughput increased as the pull rate increased until the product became hollow. (p. 72, sec. 4.7.10).

Regressions were run on these relationships (pull rate vs. pot setting, lb/in, mass throughput, product diameter and attenuation) on the average values for each pot setting as listed in Table 8, Addendum VII. The equations, R-squared values, standard deviation and T-ratio are given on p. 73, sec. 4.7.11. In each case the relationships are significant.

As run 2 progressed the product quality degraded. A blue streak started in the center of the clear cane. This became several blue streaks. The product started to become foamy in the center. Occasionally bumps were evident. Finally the

product became hollow. An X-ray tracing of the blue streak shows the product to be Ti-enriched. This is consistent with the EPR analysis performed in foamy glass remaining in the crucible at the end of Run 1.

As samples were being prepared for the CTE analysis (sandwich seals), it was found that the outer surface of the product devitrified during annealing. Surface analysis (with energy dispersive X-ray analysis or EDX) reveals the presence of Na, K, Ca, Cl and S. A one minute acid etch in hydrofluoric acid after cleaning the cane with xylene eliminated devitrification problems during annealing.

CTE results are discussed in detail in reports by H. Hagy. The average expansion of the feestock was -8 ppb/°C. The standard, composed of the same glass as the feedstock charge, average -6 ppb/°C. The observed was -4 ppb/°C, an excellent agreement with the expected result of -2 ppb/°C.

3.5.4 Conclusions

CTE

CTE results are discussed in two separate reports by H. Hagy. These reports conclude:

- A. The average for run 2 was -4 ppb/°C.
- \underline{B} . CTE is a constant, preserved in remelting and drawing.
- <u>C</u>. Blue streak material imparts a more positive expansion to the glass.
- D. Expansion variability observed is the result of compositional variability (and not the result of the downdraw process itself.)

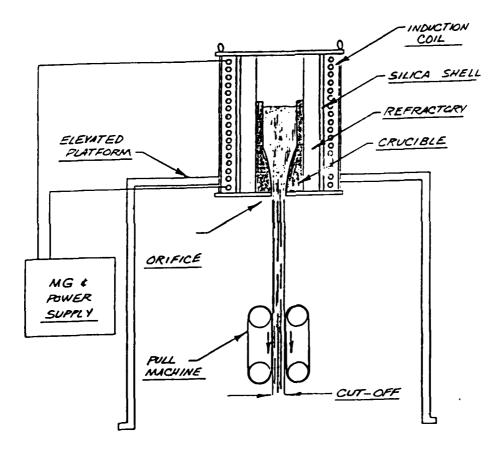
Reports by H. Hagy are attached on pp. 74-89, sec. 4.8.

Equipment

The equipment installed for Phase I performed satisfactorily and should be suitable for Phase II trails.

Given the observed attenuation ratio of 3 to 4 in Run 2, this furnace will probably not be large enough to draw strip wide enough to yield product with a 6.6" width.

DRAW



TO: Mr. P. M. Smith

cc: Mr. D. L. Prusha

FROM: W. E. Siebold/223-6802 Small Tanks & Heat Treat

Furnace Engineering Melting Technology

DATE: February 24, 1984

SUBJECT: 100 KW Motor-Generator Set, Canton Plant

This memo is intended to review our conversations, work to date, and recommended future actions concerning the 100 KW, 3000 HZ motor-generator set. My first visit to Canton was on Wednesday, 2/1. The following actions were taken:

- The removal of some external control and interlock wiring to restore the control circuit to an "as supplied" condition by Inductotherm.
- 2. M-G set appeared to be in good condition.
- Much evidence of the 1972 flood mud remains inside the enclosure.
- At least two meters are not working. Recommended that all six meters be replaced. New metering on order.
- M-G set turned over and then brought up to speed. Bearings look and sound in good shape. No cooling water on.
- T.C. field excitation tested. Approximately 100 volts open circuit generator volts obtained. Inductor coil was not connected. No cooling water on.
- Inductor coil inspected. I felt that we could load into the coil (see below for negative results).
- CGW safety people consulted by you concerning PCB insulated capacitors. May use them until 1988 as there are below 3+ pounds in each can.
- Measured for Parker fittings to equate No. 12 and No. 16 fittings so water cooled inductor coil leads could be connected to the work coil.

100 KW Motor February 24, 1984 Page 2

My second visit was on Wednesday, 2/15, with the following work done:

- Connected water circuits and tested system. Once circuit
 was not flowing, it was back flushed and then split into two
 circuits. The hoses were noted to be old and brittle.
 Their recommended replacement is underway.
- 2. Upon arrival of the Inductotherm serviceman, the M-G set was started and attempts made to load into the coil without success. Minimum generator voltage at excessive generator current was observed. M-G set was shut down and one 10.2 microfarad capacitor was added.

M-G set restarted and loaded into coil at approximately 50 volts at 140 amps at the generator. PF meter not working.

The coil appeared to have a minimum inductance causing relatively high current at low voltage to flow. Insufficient capacitors were on hand to swinge the load to unity power factor.

3. Ground detector circuit was tested and found not working. This subject should be examined carefully as an induction melting glass furnace may not need this circuit for protection. It also may be electrically difficult to connect in a meaningful manner.

The following recommendations have been mutually made and discussed with action programs listed:

- Because the generator metering is the source of information for such future systems and three meters out of six are bad, a total new set is recommended. This set is now on order. Need for installation assistance is yet to be determined.
- Because the interior of the water cooling hoses have grossly deteriorated, complete new hosing is recommended. The new hoses are now being installed.
- The existing coil is a 3 parallel turn design with low inductance. It does not appear practical to use this coil.

A new coil is recommended, 16 3/4" I.D. x 17 1/4" O.D. x 30" high with 25 to 30 turns and constructed with 1/2" O.D. copper tubing. The terminal fittings should be Parker Series 12 brass fittings to mate with the terminals on the water cooled leads.

This coil could be ordered from Inductotherm or made at the Canton plant. No work on the coil, however, should occur until it can be coordinated with the furnace design.

100 KW Motor February 24, 1984 Page 3

 Because of a 10-12 week lead time, it is recommended that the equivalent of 5-10.2 microfarad, 1250 volts, 300 KVAR capacitors be placed on order.

With a new coil design, the following tabulation indicates that the above capacitors may not be needed. Until the coil design is firmed up, we may want the protection.

Coil Turns	Max Volts At 50 KW (Work)	No. Of Cans For 200 KVAR
15	300	11.6 (12)
20	400	6.5 (7)
25	500	4.2 (5)
30	600	2.9 (3)

To use the existing coil consisting of 3 parallel 7 turn sections, I calculate that 22 capacitor cans each rated 10.2 microfarad would be required to load at unity power factor. It does not appear viable to use this coil.

 The need for new water cooled leads will depend upon your completed layout. If required, they will be ordered from Inductotherm.

I believe that the above reflects the action taken to date and the mutually discussed recommendations. Please let me know when I may be of further assistance.

JMF6/E

FUSED SILICA MELTER K-84-A CANTON, N.Y.

SPECIFICATIONS	DRAWING NUMBER
Assembly	12822-4801
DESCRIPTION	DRAWING NUMBER
Fused Silica Melter	12822-4801
Middle Bottom Plate	12822-4811
Top Bottom Plate	12822-4821
Suscepter Top	12822-1292
Lower Bottom Plate	12822-1302
Top Plate	12822-1312
Gas Distribution Tube	12822-1322
Orifice Plate	12822-1373
Muffle Plate	12822-1383
Suscepter Cover	12822-1393
Gasket	12822-1403
Susceptor Bottom	12822-1413
Pluq	EMP-F-000-71
Top Plate	EMP-QR-50
Top Plate & Plug Assy. (Ref.)	EMP-F-000-81
Bushing	24132-484A
Bushing, Isolation	24972-64A

2/84 RCB/b

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· S	Spacer						873 S	S	One		3			
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							Dwg. No. 1282	12822-4801		Unit & Plant KB	K84A	Canton	go
Rev	Item	asn	pue	DESCRIPTION		DWG. NO.	HATERIAL	REQ'D	ALLOT	r ORDER	EST	UNIT	TOTAL
	22	Condust 3/4" Abs	Aluminum Thin Wall 1/32"	in Wall			Com'l	- 7					:
	23	Bushing 3/4" Th	nk x 1 5" Dia.	Dia		24132-484A	Steatite	7					
	-7	Isolation Bushing 3/8". Thk x 1 4" Dia	ng 3/8". Ti	hk x 1 ½" Dia.		24972-64A	Steatite	5					
	55	Screw Soc. Hd C	Cup 309 Stn St. 1G	to St.1			Com'l	80					
	36 -	+	Blanket 40" Wide 11de Co. Ca	Graphite Felt Blanket Y. Tuk x 36" - 40" Wide Cat. No. X-3101 Luion Carbide Co. Carbon Products Div			Com'1	7_Yards	2	21 Yard	<u>a</u>		! ! !
		Chicago, IL 60606	60606	23									
	27	ing Parker.	#2-446-V747-75 = .275"	1-75			Fluoro- carbon	Une	!	7			1
		6660_loy_Road E. Syracuse, 1	Soad See NY 13057 8397	05.7						!			
											; 		<u> </u>

Run 1 Heat-Up Schedule	Cum. Hours
Heat 100°C/hr to 600°C	6
Hold 600°C for 4 hours	10
Heat 100°C/hr to 1650°C	20.5

Actual start-up: Tuesday, 6/5/84 at 10 am.

Ru 2 Heat-Up Schedule	Cum. Hours
Heat 100°C/hr to 600°C	6
Hold 600°C for 3 hours	9
Heat 150°C/hr to 1650°C	16

Actual Start-Up Time: Tuesday, 6/19/84, at 9 pm.

ULE DOWNDRAW FURNACE

START UP PROCEDURE

SET UP

- 8 hours before scheduled start up open the inert gas line. Set nitrogen pressure to 10. Set each flow meter to 5 1/min.
- 2. Check water filters. Replace if not clean.

START UP

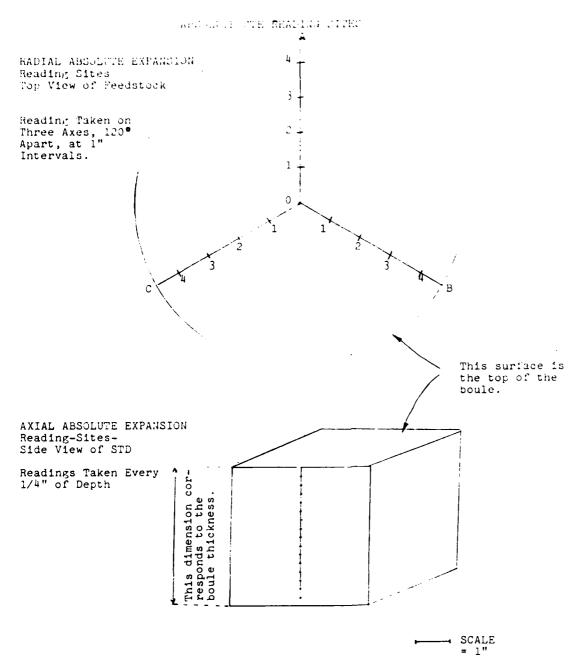
- 1. Open cooling water through filter.
- Check for adequate water outlet flow through motor generator, coil, cabinet, and plates.
- 3. Turn main power switch to on.
- 4. Set generator voltage % to zero.
- 5. Set furnace voltage control to tap 7.
- 6. Switch in numbers 1, 2, 3, 4, 5, 6 on capacitor control.
- Reset cabinet with, generator air temperature, generator overload and furnace water buttons.
- Pull starter handle down. Hold in start position for 60 seconds.
 Push starter handle through stop and into run position.
- 9. Push on generator field.
- 10. Reset starter interlock.
- 11. Push on main line contactor.
- 12. Turn up generator voltage control to initial setting.

ABSOLUTE EXPANSION READINGS - DOWNDRAW FEEDSTOCK, RUN 2

			RADIAL					AXIAI.	
PIECE #		AXISA	AXISB	AXICC	AVERAGE	GRAND AV€	DEPTH	Abs exp	AVG
4	0	-9	-	-	-9		1/4	-3	-6
	1	-8	-10	-8	-9		1/2	- 5	
	2	-7	-9	-8	-8	-8	3/4	-6	
	3	-6	-9	-7	- 7		1	-5	
	4	-4	-8	-8	- 7		1 1/4	-3	
							1 1/2	-3	
5	0	-9	-	-	-9		1 3/4	-2	
	1	-8	-8	-9	-8		2	-1	
	2	-7	-8	-8	-8	-8	2 1/4	0	
	3	-7	-8	-8	-8		2 1/2	-3	
	4	-6	-7	-8	-7		2 3/4	STRIEA	
							3	-13	
6	0	-9	-	-	-9		3 1/4	-12	
	1	-9	-9	-9	-9		3 1/2	-11	
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	3	-8	-9	3-	-8		4	-10	
	4	_7	_9	_9	-8		4 1/4	-9	

ABSOLUTE EXPANSION READINGS - DOWNDRAW FEEDSTOCK, RUN 1

		1	RADIAL					AXIAL	
PIECE #		AXISA	AXISB	AXISC	AVERAGE	GRAND AVERAGE	DEPTH	ABS EXP	AVG
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	2	-4	-4	-6	-5	-4	3/4	-6	
	3	-4	-3	-7	-5		ì	-10	
	4	-4	0	-7	-4		1 1/4	-15	
							1 1/2	-8	
2	0	+7	-	-	+7		1 3/4	-5	
	1	+6	+6	+6	+6		2	-3	
	2	+5	+4	+4	+4	+4	2 1/4	-2	
	3	+3	+2	+3	+3		2 1/2	-3	
	4	+2	+2	-1	+1		2 3/4	-5	
							3	-6	
3	0	-8	-	-	-8		3 1/4	-4	
	1	-7	-7	-7	- 7		3 1/2	-4	
	2	-6	-7	-7	-7	-6	3 3/4	-8	
	3	-7	-5	-7	-6		4	-7	
	4	-6	-3	-6	-5		4 1/4	-2	



NOTE: Axial readings of standard and feedstock should be equal since they are from the same boule.

FEEDSTOCK DIMENSIONS AND WEIGHT

Run	BOULE	PIECE NO.	DIAMETER	THICKNESS	WEIGHT	POSITION IN FURNACE
1	94-XXX	1	9.49"	4.426"	25.0 lb	вот
		2	9.50"	4.426"	25.0	MID
		3	9.49"	4.433"	25.0	TOP
		TOTAL		13.285" a	75.0 lb b	
2	34-109	4	9.49"	4.422".	25.0	BOT
		5	9.49"	4.421"	25.0	MID
		6	9.48"	4.422*	25.0	TOP
		TOTAL		13.265" ^a	.75.0 lb b	•

a charge height

b charge weight

- W	GENERATOR CFERATION	EKATION						CAFACTUR CONTROL (In-Out)	MTKQ	In-041				-	ANTER FLOW	3			TEMPERATURE, C.	uae, c.
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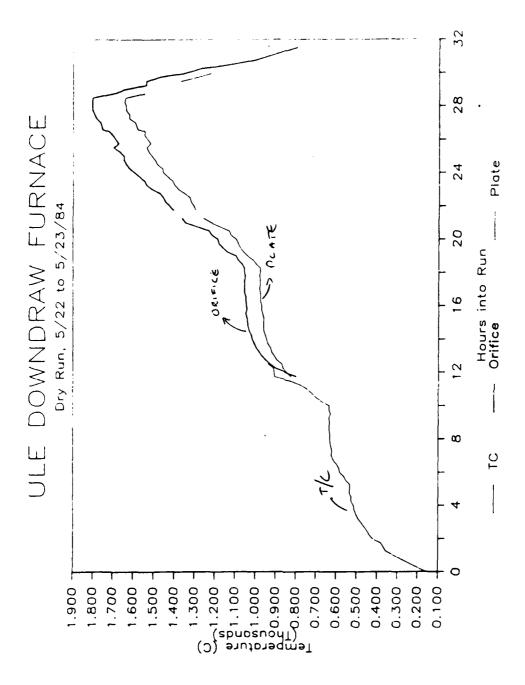
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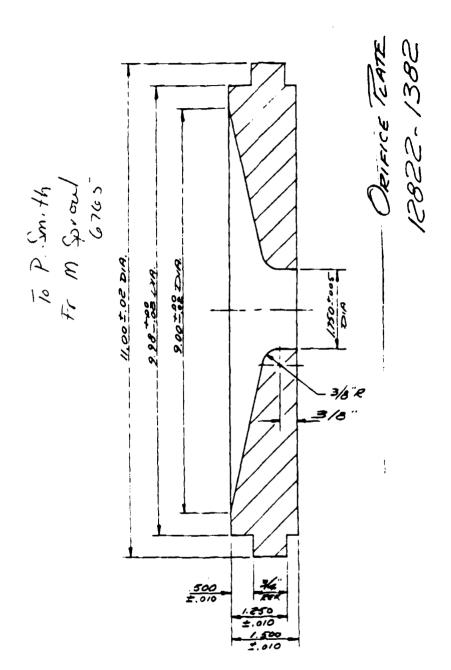
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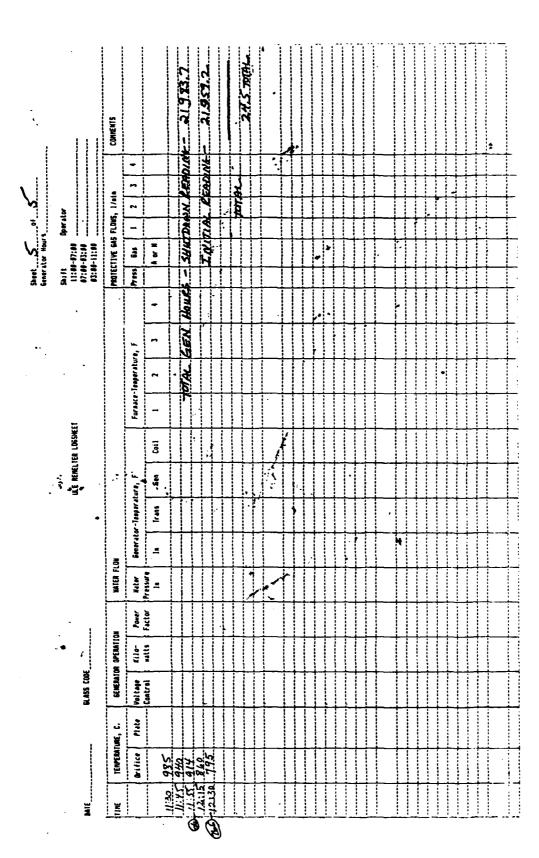
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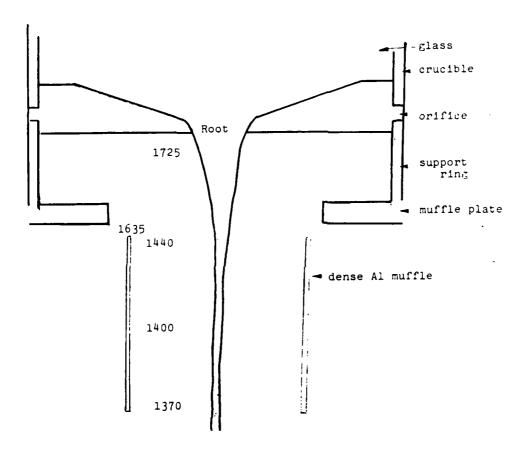
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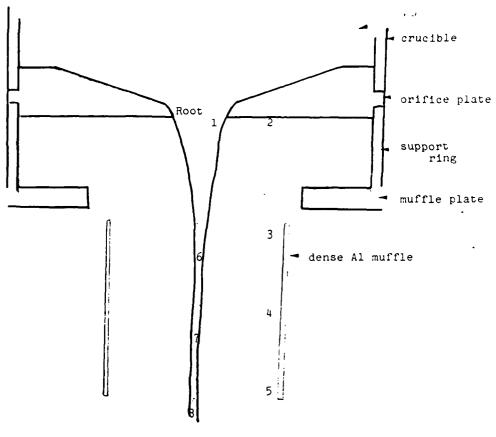
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Temperature Readings at Furnace Bottom; Run 1 6/6/84 10 a.m.



Temperature Readings at Furnace Bottom; Run 2 6/20/84



Time	1	2	LOC 3	ATION 4	5	6	7	8
0850	1691	1732	1606	1373	1330	1452	1339	959
0937	1742	1662	1589	1495	1290	1454	1246	971
1021	1747	1657	1620	1518	1401	1453	1317	1185

ULE DOWNDRAW FURNACE

SHUTDOWN PROCEDURE

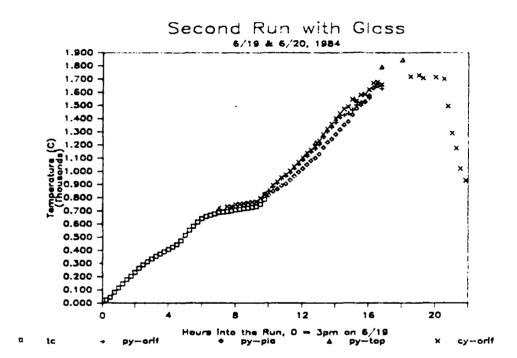
- Turn generator voltage control to zero percent.
- 2. Shut of main line contactor.
- Shut off generator field.
- 4. Push and hold motor generator starter button until starter relay drops put (about 10 seconds).
- 5. Leave gas and water on to cool furnace.

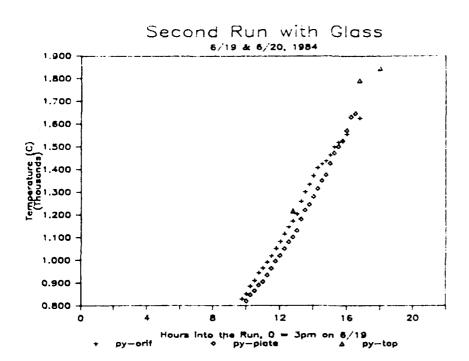
Gas Settings

Nitrogen pressure at 10 Set each flow meter to 51/min Shut pressure and flow off after 4 hours.

Water

Leave water on till furnace is cool. (12 hour minimum).





H. E. Hagy Corning Glass Works

1. Introduction

A block of ULE^m titanium silicate 6" x 6" x 4" was selected for study. Three 2" diameter x 4" long cylindrical slugs were core drilled from this block and induction remelted for ½ hour at 1650°C. on three separate furnace runs. Following these remelting thermal cycles, chosen to simulate remelting for redrawing, all three were annealed together on the ULE^m standard anneal.

The purpose of exposing ULE™ to remelt thermal conditions is to determine if any significant change occurs in key physical properties. Large or erratic changes may influence decisions on proceeding with the remelting and drawing facility at Canton.

2. Sample Plan

From each of the three cylindrical slugs these pieces were cut for the purpose stated:

- (a) a 1/8" x 3 7/8" x 1 7/8" piece to be fritted to two identical parts from the original block to form a symmetrical sandwich seal.
- (b) a 3/8" diameter x 4" rod for superdilatometer 5 to 35°C. thermal expansion coefficient. An identical rod was taken from the original block as well.
- (c) a 1/10" x 1/10" x 3" beam for beam bending annealing and strain point. An identical beam was taken from the original block.

3. Experimental Results

3.1 Symmetrical Sandwich Seals

Through photoelastic analysis the symmetrical sandwich seal yields the difference in expansion from the setting point (925°C) to 25°C. No significant differences are found.

Cylindrical Slug No.	Block Minus Slug Aa, ppb/ ^O C					
1	+ 0.36					
2	+ 4.65					
3	- 0.90					

REPORT

EVALUATION OF THERMAL EXPANSION AND VISCOSITY CHANGES IN ULE TITANIUM SILICATE DUE TO THERMAL HISTORY SIMULATION OF REMELT FOR REDRAW.

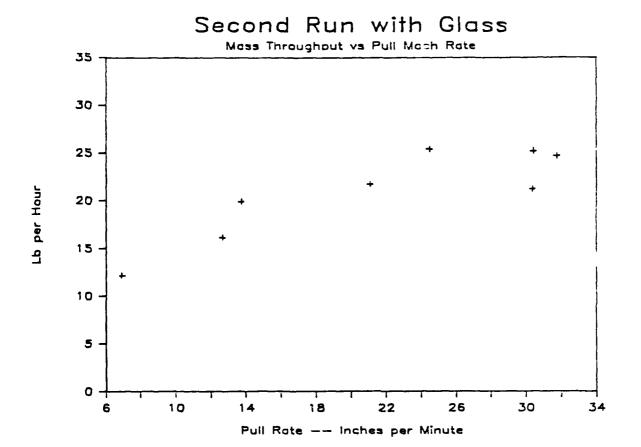
Contract No. F30602-83-C-0121

Corning Glass Works
Research & Development Division

September 28, 1983

Run 2 With Glass - Linear Regressions

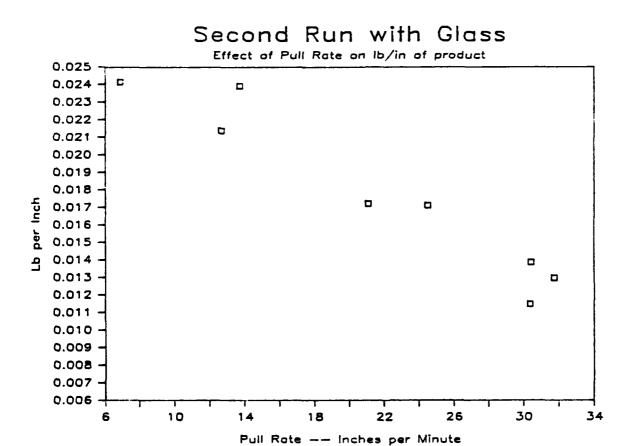
	<u>T-ratio</u>	Std. Dev.	R ² Value
Pull Rate = 4.43 + .441 (pot setting)	-	3.359	87.3%
Pounds In = .02840005 (pull rate)	-7.83	.001598	89.6%
Mass Thru = 11.6 + .433 (pull rate)	4.38	2.463	72.2%
Prod. Diameter=.7340088 (pull rate)	-13.27	.01646	96.2%
Attenuation = 2.24 + .0484(pull rate)	11.94	.1010	95.3%



Second Run with Glass Effect of Pull Rate on Product 4.0 3.5 3.0 2.5 Product 2.0 1.5 1.0 0.5 0.0 6 10 18 22 30 34 26 Pull Rate —— Inches per Minute diameter (in) A attenuation

Second Run with Glass Product Diameter vs Pull Rate 0.700 0.680 0.660 0.640 0.620 Product Diameter (in) 0.600 0.580 0.560 0.540 0.520 0.500 0.480 0.460 0.440 10 18 22 26 30 34

Pull Rate -- Inches per Minute



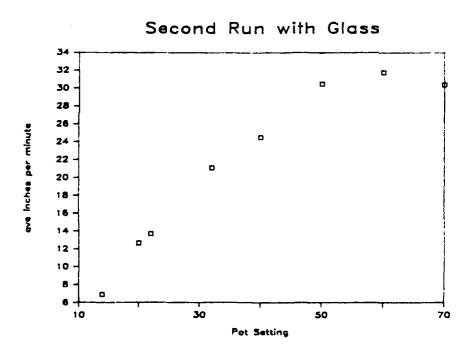
Pull Rate (in/min)

Pot Setting	Calculated	Timed	Average
14	-	6.9	6.9
20	14.2	11.1	12.7
22	14.7	12.7	13.7
32	22.2	20.0	21.1
40	26.7	22.2	24.5
50	32.0	28.9	30.4
60	31.5	31.7	31.7
70	28.8	31.9	30.7

Second Run with Glass

pot	Ave	Ave	Ave	Ave	Ave
setting	lb/in	in/min	thruput	diameter	atten
			(lb/hr)	(in)	
14	0.024	6.9	12.2	0.682	2.6
20	0.021	12.7	16,2	0.637	2.8
22	0.024	13.7	19.9	0.604	2.9
32	0.017	21.1	21.8	0.526	3.3
40	0.017	24.5	25.4	0.513	3.4
50	0.014	30.4	25.2	0.481	3.6
60	0.013	31.7	24.7	0.474	3.7
70	0.011	30.3	21,2	0.455	3.9

Page 37

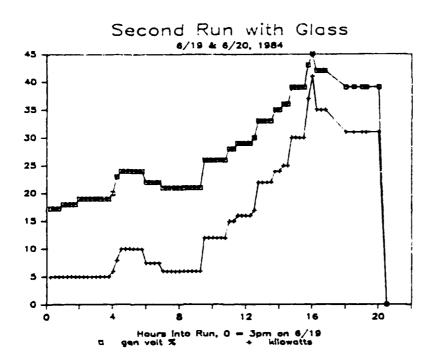


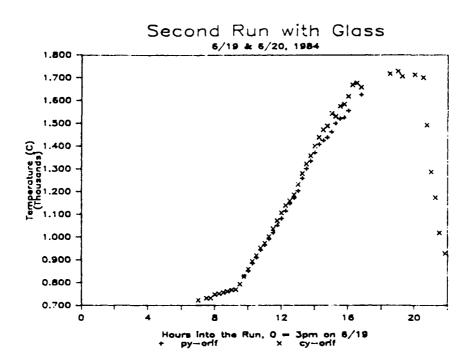
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14 6 2.95					0.573	:. : £
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57	1201	:80	1.43	<u>:</u> !		0.479	7.65
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22	1012	200					
ŧ?	:017	202	2.44	35		0.452	1.27
76	-1014	187	9.41	??		0.455	2.74 · *
7è	1016	204	0.45	. 7		0.479	1.45
79	1917	275	0.50	7.		0.471	7.72 -
?:	1015	211	0.45	37	•:	0.475	7.::
72	1019	216	0.48	37		0.453	7. EF 4
72	1020	200	2,44	25		0.494	7.49
74	1021	193	0.44	36		1.481	1.64
							1.30
75	1027	190	9.42	34		0.4±1	
75	::::4	:::	1.45	37		0.490	7.17
7*	1576	:55	2.41	17		475	3.:0
72	1:25	151	9.32	34		0.420	4.17 °; buep
79	1324	:97	0.42	43		2,425	4.12 * : sump
90	1029	:94	2.41	38		2.42:	4.16 , busp
8:	1032	170	1.37	37		0.411	4.27 blue; very busts,
9:	1478	167	0.75	34			4.15
ěť	1039	242	3.75	75	22 1		
_		-	0.74		•.		•
29	1942	32?	3.74	23			
84	1045	334	C.74	34			
97	:048	7:2	0.69	32		0.:40	2.73 blue; foar & bueps
97	1052	405	0.59	41			•
28	1023	355	0.78	36		0.619	2.63
99	1059	332	0.72	35		0.637	2.75 *
92	1101	371	0.92	39		0.600	2.91 blue: foak & risples
91	1106	399	0.98	41		0.639	2.74
97	1109	42.	0.93	42		0.536	2.75
93	1111	401	3.89	4!		J. 657	2.55 tlue; foar to moile.
94							
	1112	214	0.70	35		0.655	2.63 tiue; hollow
95	11:4	242	0.53	34	5¢	9.666	2.67
96	1116	:50	0.35	37		0.461	2.80 .
97	1117	157	2.35	36		0.517	2.38 .
98	1:18	195	0.43	35		0.475	3.68 *
99	1119	195	0.43	45		0.485	3.51 *
100	1120	172	0.38	28		0.503	3.40 ~
101	1121	218	0.49	48		0.515	3.40
102	1122	168	0.37	45		0.478	3.66
103	1:23	126	0.28	41		0.455	3.85
105		110	0.24	46			
	1124					0.440	3.98
105	1125	80	C. 19	38		0.445	3.93 dark; hollow
196	1:26	92	0.20	, 45		0.430	4.07
:07	1:27	8¢	C.18	42		0.455	3.95 •

CANE MERELEPTINGS-SECTION RUN N. SLAST

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:5	340	:22	1.71	7.1		0.519	2,95 '
:7	346	322	1.71	: :		2.6.9	2.53
:8	548	120	. 10	7:		1.612	1,54
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24 22	904	586	. 20	13 4£			7.45 7.45
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76	612		(.74	7,4	::		5.15
	5:5	275		75		:. ::-	3,26
:3	917	244	1.53	7.7		3.510	3.31 clear
<u>:</u> ;	213	252	3.58	34			1 49
: ::	826	220	3.49	29			7.75
-	322	226	€.56	74		1.514	1,47
::	977	157	7	27		. 5 . 4	3.29
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34	127	290	3.24	19		575	1.00 payet
75	52¢	252	7.54				7 (5 0) - 00000
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40	415	216	¢. ≠9	29			3.35
4:	743	244	2.54	3 1		v. 5.	J.26 °
42	94;	302	1.67	:8		0.575	3,27
42	945	760	:.57	:2			7.26 several blue streams
44	764	248	1.55	31		0.536	3.25
45	-4 <u>5</u>	392	4.26	37	41		3.28 *
16	747	234	9.52	Ĭ.			3.29
47	346	272	0.40	:4		1.517	3.26
	350	244	0.54	::			3.50
48		_				0.499	
49	:5!	242	j. .	33		(,4%	3.53
50	657	772	0.51	2.		0.502	3.49 *; tuap
51	954	226	9.29	33		0.503	3.49 *





3.2 Beam Bending Viscometry

Measurement of annealing and strain points provides a sensitive indication of composition and/or structure change. No significant differences are indicated.

	Annealing Point, OC	Strain Point, OC
Slug No. 1	1008	897
Slug No. 2	1008	898
Slug No. 3	1009	899
Original Block	1007	896

3.3 Superdilatometer

The superdilatometer has a standard deviation of $\pm 5 \text{ppb}/^{\text{O}}\text{C}$ and is much less precise than the sandwich seals. Results are:

Cylindrical Slug No.	Block Minus Slug, ppb/OC
1	+ 2
2	+18
3	- 6

There is a slight suspicion that a real difference exists between the block and slug #2. Sandwich seals show this also.

4. Conclusion

The simulated remelt thermal cycle has negligible effect on these properties of ULE m titanium silicate. Only slug #2 shows a small expansion difference which may well have been in the starting composition.

TEST REPORT

"Evaluation of Thermal Expansion
Change in ULE™ Titanium Silicate
Following Remelting and Downdrawing Phase I, Run 2"

H. E. Hagy July 24, 1984

1. Introduction

Phase I, Run 2 of the remelting, downdraw program at the Canton plant produced cane ranging from \(\frac{1}{2} \)" to 3/4" in diameter. A key question about this process is whether the thermal expansion of the ULE glass is altered.

Accordingly, a sandwich seal testing program was developed through the cooperative efforts of Canton and R & D personnel to answer this question. Through the use of a "standard" ULE" of nearly the same original expansion, but not remelted, downdrawn specimens can be compared by frit sealing. Adequate sensitivity is provided at a cost far less than direct expansion measurements.

2. Standard and Downdraw Glass

2.1 Standard

The pieces representing standard unmelted glass were cut from a block, Piece #8, described on Figure 1. Expansion values on this figure are ultrasonically derived and represent the 5 to 35°C coefficient in units of ppb/°C. Standard pieces for sandwich seals, 0.080" x 0.45" x 1.5", were cut from the shaded area. Since locations of individual pieces were not documented, it is obvious from the x-axis plot that the standard has a range of expansion of 7 ppb/°C.

2.2 Downdraw Specimens

Figure 2 shows how downdraw pieces, with the same dimensions as the standard pieces, were cut from the drawn cane. Nine downdraw cane specimens described in Figure 2 were selected for study. One piece for sealing was cut from each of these nine canes.

Radial expansion profiles for the three $9\frac{1}{2}$ " diameter discs stacked vertically to make the furnace charge are shown in Figure 2A.

2.3 Annealing

All parts used in the sandwich seal testing were given the standard ULE $^{\rm m}$ fine anneal to normalize thermal history.

3. Sandwich Seals

Spaced sandwich seals were fritted in the geometrical configuration shown on Figure 3. U-type frit, M-14-3504, BM-82-251, was used and fired two hours at 960° C. A ridealong F-G-F wafer seal showed perfect expansion match between the frit and ULE^m.

4. Experimental Method

Following sealing, the optical retardation in the central (downdraw) seal member was measured using a Friedel polarimeter. The difference in thermal expansion coefficients between standard and downdraw from the setting point (925°C) and room temperature (25°C) is given by the following equation:

$$\Delta \alpha = \frac{\lambda A F}{180 k p E \Delta T} \left[1 + \frac{t_d}{2t_s}\right] 10^9$$
 (1)

where:

 $\Delta \alpha$ = expansion coefficient difference, ppb/ $^{\circ}$ C,

 λ = peak wavelength of filtered white light, 546 nm.

A = rotation of Friedel analyzer, degrees,

t_d = t_s = specimen thicknesses, 2.0 mm,

k = stress optical constant, 0.292 nm/cm/psi,

p = seal path length = 1.15 cm,

 $E = elastic modulus, 9.8 \times 10^6 psi,$

 ΔT = setting point minus R.T., 900°C, and

F = shape factor.

The shape factor F which accounts for spacer shear and bending is given by:

$$F = \left[1 - \frac{4}{3} \left(\frac{t}{w-2b}\right) \left\{2 \left(\frac{d}{b}\right)^3 + \frac{3}{2} (1+r) \left(\frac{d}{b}\right)\right\}\right]^{-1}$$

For this geometry, F = 1.07 (Poisson ratio r = 0.17)

Equation (1) reduces to:

$$\Delta \alpha = 1.63A$$

Our main interest is not in this $\Delta\alpha$ but in the $\Delta\alpha$ between 5 and 35°C. We have established for ULE^m that $\Delta\alpha$ is a constant for any two different specimens and applies over any temperature internal, providing thermal history is normalized. The question arises whether this holds for the downdraw vs. the standard specimens. Thus two of the sandwich seals were measured at 109°C as well as at 25°C to test this principle for the downdraw glass. Equation (1) is modified accordingly:

*A is replaced by ΔA_{\star} the change in analyzer rotation between 25 and $109^{\circ}C$

$$*\Delta T = 84^{\circ}C$$

then $\Delta\alpha' = 17.5 \Delta A$

5. Experimental Results

Resulting expansion differences are given in Table 1. All differences are in units of $ppb/^{O}C$ and are relative to the standard. Hence a negative sign signifies that the downdraw expansion is less positive than the standard.

The values for A in the table are in degrees and a letter C or T signifies that the downdraw specimen is in compression or tension, respectively.

For downdraw samples 50 and 53, the blue streaks made conventional reading of retardation impossible due to their opaque character. For these two sandwich seals the optical retardation was measured in the standards. To infer the reading in the central member, one must reverse the stress sense and multiply the retardation by a factor of two. The other seals verified this theoretical principle.

6. Conclusions

The grand average expansion of the remelt charge is $-8~ppb/^{\circ}C$. The standard material is experimentally defined as $-6~ppb/^{\circ}C$. Therefore, on the average, the expected $\Delta\alpha$ should be $-2~ppb/^{\circ}C$. Excluding sample 2, which seems to have a special problem, and samples 50 and 53, having blue streaks, the grand average $\Delta\alpha$ emerges as $-4~ppb/^{\circ}C$. Considering the variability of the standard (we used only 18 of the 39 pieces cut) and the uncertainty of the sandwich seal test, $\pm 2~ppb/^{\circ}C$, this is excellent agreement.

Sample 2 is a problem. Either the process has a start-up peculiarity or the bottom charge cylinder had a serious axial composition problem. Unfortunately no axial expansion data were taken on these charge cylinders. However we can get a chemical analysis on this piece (right from the sandwich seal) and at least answer the composition question.

For the special experimental setup that I used to determine $\Delta\alpha'$, the best ΔA uncertainty attainable is $\pm 0.5^{\circ}$ C, which imparts an uncertainty in $\Delta\alpha'$ of ± 9 ppb/°C. Therefore, in consideration of these experimental uncertainties, $\Delta\alpha$ is statistically equivalent to $\Delta\alpha'$. Apparently then, the important principle of $\Delta\alpha$ being a constant has been preserved in remelting and drawing. This is very encouraging!

Blue streak material seems to impart a more positive expansion to the glass.

TABLE 1. Expansion Differences of Downdraw Specimens

Relative to Standard

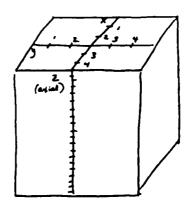
Sample ID	<u>A</u>	ΔΑ	Δα	Δα,
2	14C	-1.6	-23	-28
4	4C		-7	
8	4C		-7	
16	10		-2	
24	0		0	
33	2C	-0.1	-3	-2
50	4 T		+7	
53	6T		+10	

FIG. 1

RADIAL DOWNDRAW ABS. EXPANSION

PIECE NO 8 - Rum Z - STANDARD

RADIUS	AXIS X	AXISY	AUG
ሃ _	-8	-7 `	-6
i	-7	-8	
1/2	-8	-7	
2	-7	-7 ·	
21/2	-6	-5	
3	-5	-3	
3%	-4	-2.	
4	-1	-0	



leading taken 5/10/54 by m. Best

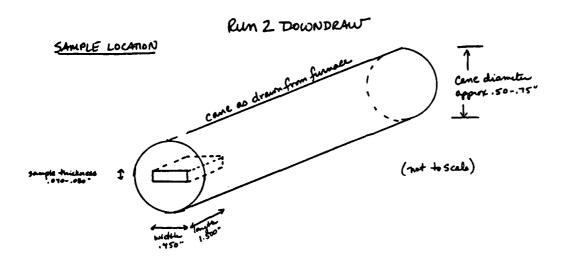
AXIAL DOWNDEAW ABS ELPANSION

PIECE NO 8 - Runz - STANDARD

Puter subjective for the subject

<u>Дертн</u> % % %	<u>abs Ex</u> p -3 -5	<u>а</u> ис- - L	leadings taken 5/0/04 h	y M.Best
h 3/,	-5 -6			
1	-5			
174	-3			7
12	-3	/5-		
134	-2 -1		(38) Siring to the state of the	1
2 2/4	_		gijustradu.	
214	م ه	#1 (F) 2	(a) Shirt	
21/2	-3		- 103-	İ
234	STRIEA	4-12"	•	Piece *
3	-13	ļ	ļ	1
3 34	-12	1		•
3%	-1/			
334	-10-	1	2000	
4	70		SCANE:	
	^ _			

Fig. 2



Sample Identification	Time Taken in Run	Description of Cane
2	0800	Clear cane
4		•
8		•
16	0843	•
21 🛊	0854	•
24	0904	Surface devit?
33	0925	Clear Cane
50	0953	Several Blue Streaks
53	0956	Blue foamy center

BROKEN UPON REMOVAL FROM PRIT PIRE PURYACE

TIG.ZA RADIAL DOWN DRAW GLASS ABS, EYPANSION 5/2/94 244 RUN

Piece# 4 mik. 4.42

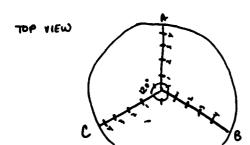
RADIUS	AXIS A	AXISB	AXISC	AVG.	GRAND AVE
0	-9			-9	
1	-8	- 10	- 8	- 9	
2	-7	- 9	-8	- 8	-8
3	-6	-9	- 7	-7	
4	-4	-8	-9	-7	

Piece # 5 Thick 4.42"

RADIUS	AYIS A	AXIS B	AXIS C	AVG	- GRAND AVL
٥	-9	_	-	- 9 ⁻	
ì	-8	- 8	-9	- 8	
2	- 7	-8	- 8	-9	-8
3	-7	-8	-8	-8	
4	-6	- 7	- 8	- 7	
•		-			

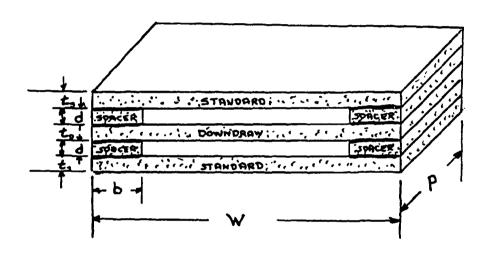
Piece # 6 Thit 4.42"

RADIUS	AXIS A	AXIS B	Alis C	AVG	GRAND AUG
0	-9.	-	-	- 9	-
1	-9	- Ŷ	-9	– ġ	
2	-9 .	- ġ	- 9	- 9	<u> -9</u>
. 3	- 8	<u>- 9</u>	-8	-9	•
4	-7	<u>-</u> ġ	-9	- 8	



NOTE: These are the readings from the three 91/2"dientes dies placed in the furnace for run 2 Each disk weighed 25 St.

FIG. 3 SPACED SANDWICH SEAL GEOMETRY



NOTE: HERVY LINES REPREJENT

To: Ms. Deborah Foss - Canton

cc: Mr. E. T. Decker
Dr. A. M. Filbert
Mr. E. H. Fontana
Mr. H. G. Freeland
Mr. W. C. Lewis
Mr. C. E. Peters
Mr. C. L. Rathmann
Mr. K. J. Schmidt
Mr. P. M. Smith
Mr. N. D. VanDyke

From: H. E. Hagy Date: August 3, 1984

Subj: Downdraw Sample 2

As suggested in my conclusions in the subject report of July 26, chemical analyses were made on both standard and sample 2. The report is attached.

The difference is wholly explained by ${\rm TiO}_2$ differences as one can see from my scribbling. Thus, it seems logical to exonerate the process.

/jt Attachment

TTER: 00 00 00 00 00 00 00 00 00 00 00 00 00			
07/31/84	PROJECI NAM SPECTRO	E 7971 OR 918A 5349- 5349/84 E: ULE DOWNDRA: GQ-84-103	<u>-</u>
E DESCRIPTION		· · · · · · · · · · · · · · · · · · ·	
84= CODE .7971; SEAL TEST .STANI 84= SAME PHASE I; RUN Z	DARD	No. 2	
	· 	· · · · · · · · · · · · · · · · · · ·	=
SE			
ECTED EXPANSION DIFFERENCE DUP NS FOR CHEMICAL COMPOSITION TO EN STANDARD AND DOWNDRAW			
NED TO ANALYSIS DESIRED	ANALYST NO	DATE REPORTED	
SPEC	ANP-1148	7/31/84	
TI	CSW-195	7/30/84	
E REPORTED 8/1/84 jm 5	REPORT SENT TO:	RAB, JSS	
CARD NO. %Ti	02	more pirul	mi
5348 6.9	of [5+2] (Hz] e	unto be high a	game-i-
53497.3	1 [doundraw]	unto the turn and acqueen with so	M 790))
	-		
			
pec attached $(7.31 - 7.6)$	6.99) × 0.5=	0.022 ppn)00	
	2, 1- 22,	= 22 ppb/eg.	*
· · · · · · · · · · · · · · · · · · ·	<i>h</i>	acquitade agrees	_ -

REPORT SHEET SPECTROGRAPHIC SEMI-QUANTITATIVE ANALYSIS

Spectro Plate No. GQ-84-103 Analyst No. ANP-1148		Analysis	Spec		
		ANP-1148	Date	7/31/84	
	dure & Commen		_		
					•
	· .			· /	
	Std	Sample			
	5348-84	5349-84			
	Si	Si			
30%					
10%	Ti	Ti			
3.0%					
1.0%					
0.3%					
0.18					
0.03%					
18	Al .	Al			
	al lower deter	mimation limit	. fon -11 + h	- alamanta	
	ned are:	dination limit	es for all the	e elements	
EXQUIL!	ien die:				•
					
•					
	K,P				
•	As,Cd				
31	Nb, Zn				

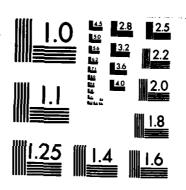
Sb,Si,Sn,Sr,Ti,V,Zr

Ag, Al, B, Ba, Be, Bi, Ca, Co, Cr, Cu, Fe, Ga, Ge, In, Mg, Mn, Mo, Na, Ni, Pb,

DOWNDRAW EXTRUSION OF ULE(TM) GLASS(U) CORNING GLASS MORKS CANTON NY P M SMITH ET AL. DEC 84 RADC-TR-84-259 F38682-83-C-8121 UNCLASSIFIED F/G 11/2 NL

2/2

AD-A153 783



MICROCOPY RESOLUTION TEST CHART NATIONAL BUREAU OF STANDARDS-1963-A

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Rome Air Development Center

RADC plans and executes research, development, test and selected acquisition programs in support of Command, Control Communications and Intelligence (C³1) activities. Technical and engineering support within areas of technical competence is provided to ESP Program Offices (POs) and other ESP elements. The principal technical mission areas are communications, electromagnetic guidance and control, surveillance of ground and aerospace objects, intelligence data collection and handling, information system technology, ionospheric propagation, solid state sciences, microwave physics and electronic reliability, maintainability and compatibility.

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